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Effect of Operational Parameters for Removal of Fluorescein Sodium Dyes by Bentonite

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Abstract: *The removal of synthetic dyes from industrial wastewater has become a critical environmental concern due to their toxic and non-biodegradable nature. This study investigates the parameters governing the adsorption of Fluorescein Sodium dye onto bentonite clay. Batch adsorption experiments were conducted at varying temperatures (298, 308, 318, and 328 K), initial dye concentrations (10-100 mg/L), contact times (30-240 minutes), and pH values (2-10). The maximum adsorption capacity was found to be 85.2 mg/g at 298 K at pH 6. Thermodynamic parameters including Gibbs free energy change (ΔG°), enthalpy change (ΔH°), and entropy change (ΔS°) were calculated using Van't Hoff equation. The negative ΔG° values (-15.8 to -18.7 kJ/mol) confirmed the spontaneous nature of the adsorption process. The positive ΔH° value (+12.4 kJ/mol) indicated endothermic adsorption, while positive ΔS° (+95.2 J/mol·K) suggested increased randomness at the solid-liquid interface. Kinetic studies revealed that the adsorption followed pseudo-second-order kinetics, and equilibrium data fitted well with Langmuir isotherm model. The results demonstrate that bentonite is an effective and environmentally friendly adsorbent for Fluorescein Sodium dye removal from aqueous solutions.*

Keywords: Operational Parameters; Adsorption; Bentonite; Fluorescein Sodium; Water treatment

I. INTRODUCTION

The textile and cosmetic industries generate colored wastewater containing dyes that poses significant environmental challenges. Synthetic dyes are particularly problematic due to their complex aromatic structures, high stability, and resistance to conventional biological treatment methods. Fluorescein Sodium, a widely used fluorescent dye in various industrial applications, exhibits high solubility in water and can cause aesthetic pollution and potential health risks when discharged into aquatic systems.

Among various treatment technologies, adsorption has emerged as a low - cost and efficient method for dye removal due to its natural abundance, high removal efficiency and potential for adsorbent regeneration. Natural clay minerals, particularly bentonite, have gained attention as promising adsorbents due to their abundant availability, large surface area, high cation exchange capacity, and environmental compatibility. Bentonite, primarily composed of montmorillonite, possesses unique structural properties including high specific surface area (20-50 m^2/g), excellent swelling capacity, and strong adsorption potential. The layered structure of bentonite provides numerous active sites for dye molecule interaction through various mechanisms including electrostatic attraction, van der Waals forces, and hydrogen bonding.

Understanding the thermodynamic aspects of adsorption is crucial for optimizing process parameters and predicting the feasibility of adsorption systems at industrial scale. Thermodynamic parameters provide insights into the spontaneity, heat effects, and structural changes occurring during the adsorption process. Despite numerous studies on dye adsorption using clay materials, comprehensive thermodynamic investigations specifically focusing on Fluorescein Sodium removal by bentonite remain limited.

This study aims to evaluate the thermodynamic feasibility of Fluorescein Sodium dye removal using bentonite clay and to determine the optimal operating conditions for maximum removal efficiency. The research contributes to the development of sustainable wastewater treatment technologies and provides fundamental understanding of clay-dye interactions.

II. METHODOLOGY

2.1. Materials : Fluorescein Sodium dye was obtained from Loba Chemie Pvt. Ltd. Bentonite clay was sourced from a local supplier and purified by removing impurities through washing with distilled water and drying at 110 °C for 24 hours. All chemicals were of analytical grade, and NICE demineralised water was used throughout the experiments.

2.2. Batch Adsorption Experiments : Batch adsorption experiments were conducted in 250 mL Erlenmeyer flasks containing 100 ml of dye solution and predetermined amounts of bentonite. The flasks were agitation in a temperature-controlled shaking incubator at 220 rpm. The effects of parameters were studied:

- Temperature : 298, 308, 318, and 328 K
- Initial dye concentration : 10, 20, 40, 60, 80 and 100 mg/L
- Contact time : 30, 60, 90, 120, 150, 180, and 240 minutes
- pH : 2, 4, 6, 8 and 10 (Adjusted by HCL and NaOH)
- Adsorbent dosage : 0.5, 1.0, 1.5, 2.0, and 2.5 g/L

After equilibrium, samples were centrifuged at 4000 rpm for 10 minutes, and the supernatant was analyzed for residual dye concentration using UV-Vis spectrophotometry at λmax = 490 nm.

2.3. Analytical Methods : The concentration of Fluorescein Sodium was determined using a UV-Vis spectrophotometer (Systronics, Double Beam- 2203) at maximum absorption wavelength of 490 nm. A calibration curve was prepared using standard dye solutions in the concentration range of 1-100 mg/L ($R^2 = 0.999$). The amount of dye adsorbed (qe) and removal efficiency (%) were calculated using the following equations:

$$qe = (Ce - Ce) \times V / m \quad ..... (1)$$

$$\text{Efficiency (\%)} = (Co - Ce) / Co \times 100 \quad ..... (2)$$

Where, Co and Ce are initial and equilibrium dye concentrations in mg/L
V is the volume of solution (L),
m is the adsorbent mass in g.

2.4. Thermodynamic Analysis: Thermodynamic parameters were calculated using the

Van't Hoff equation : $\ln(Kd) = -\Delta H°/RT + \Delta S°/R$ ..... (3)

Where Kd is the distribution coefficient (qe/Ce),
R is the gas constant (8.314 J/mol·K)
T is the absolute temperature (K).

The Gibbs free energy change was calculated using: $\Delta G° = -RT \ln(Kd)$ ..... (4)

III. LITERATURE REVIEW

3.1. Dye Pollution and Environmental Impact : Industrial dye pollution has become a global environmental concern, with an estimated 10-15% of dyes being lost during manufacturing and processing operations. Textile industries discharge approximately 2-3 million tons of synthetic dyes annually, causing severe water pollution and ecosystem disruption. The persistent nature of synthetic dyes in aquatic environments poses long-term risks to human health and marine life .

3.2. Adsorption Mechanisms on Clay Materials : The adsorption of organic dyes on clay minerals involves complex interactions governed by surface chemistry and molecular properties. Montmorillonite unit exhibit excellent adsorption capacity for cationic dyes through electrostatic interactions and ion exchange mechanisms. Surface functional groups in dye-clay interactions, highlighting the importance of OH and Silicon in the adsorption process.

3.3. Thermodynamic Studies of Dye Adsorption : Thermodynamic parameters provide information about the nature and feasibility of adsorption processes. Thermodynamics of methylene blue adsorption on various clay materials and found that negative Gibbs free energy indicated spontaneous adsorption. Similarly, researcher reported endothermic adsorption of Congo red on bentonite with positive enthalpy and entropy changes.

3.4. Bentonite as Adsorbent : Bentonite has been extensively studied for adsorption capabilities due to it is easily available in the nature. Natural bentonite could achieve >90% removal efficiency for various dyes under optimized conditions. The high surface area and cation exchange capacity of bentonite make it an ideal adsorbent for environmental remediation applications.

3.5. Kinetic and Equilibrium Studies : Adsorption kinetics and equilibrium behavior is essential for process design and optimization. Recent studied showed that pseudo-second-order kinetics best described dye adsorption on clay materials, indicating chemisorption as the rate-limiting step. Equilibrium studies typically isotherm models, providing insights into adsorption capacity and mechanism.

IV. RESULTS AND DISCUSSION

4.1. Adsorbent Characterization : The BET surface area of bentonite was determined to be 42.3 m^2/g with a pore volume of 0.089 cm^3/g. XRD analysis confirmed the presence of montmorillonite as the major mineral phase with characteristic peaks at 2θ = 5.8°, 19.8°, and 35.2°. SEM images revealed a layered structure with irregular particles ranging from 0.5-5 µm in size. The cation exchange capacity was found to be 85.6 meq/100g, indicating good potential for dye adsorption.

4.2. Effect of Operating Parameters

4.2.1. Effect of Contact Time and Temperature : Table :1 description of the experimental observations for the effect of contact time on Fluorescein Sodium adsorption at different temperatures.

Table 1: Effect of Contact Time on Dye Removal at Different Temperatures (Initial dye concentration: 50 mg/L, Adsorbent dosage: 1.5 g/L, pH: 6)

Contact Time (min)	Temperature (K)	Ce (mg/L)	qe (mg/g)	Removal Efficiency (%)
30	298	32.5	11.7	35.0
60	298	18.4	21.1	63.2
90	298	12.8	24.8	74.4
120	298	9.2	27.2	81.6
150	298	7.6	28.3	84.8
180	298	7.1	28.6	85.8
240	298	7.0	28.7	86.0
30	308	28.9	14.1	42.2
60	308	15.2	23.2	69.6
90	308	10.8	26.1	78.4
120	308	8.1	27.9	83.8
150	308	6.8	28.8	86.4
180	308	6.2	29.2	87.6

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240	308	6.0	29.3	88.0
30	318	26.1	15.9	47.8
60	318	13.5	24.3	73.0

4.2.2. Effect of Initial Dye Concentration :

Table 2 shows the experimental data for different initial dye concentrations at 298 K.

Table 2: Effect of Initial Dye Concentration on Adsorption (Temperature: 298 K, Contact time: 180 min, Adsorbent dosage: 1.5 g/L, pH: 6)

Initial Concentration (mg/L)	Ce (mg/L)	qe (mg/g)	Removal Efficiency (%)
10	1.2	5.9	88.0
20	3.1	11.3	84.5
40	7.8	21.5	80.5
60	13.2	31.2	77.7
80	20.1	39.9	74.9
100	28.5	47.7	71.5

4.2.3. Thermodynamic Analysis :

Table 3 : Represents the thermodynamic parameters calculated from the adsorption data.

Table 3: Thermodynamic Parameters for Fluorescein Sodium Adsorption

Temperature (K)	Kd (L/g)	ln(Kd)	ΔG° (kJ/mol)
298	4.03	1.394	-15.8
308	4.71	1.550	-16.4
318	5.51	1.707	-17.1
328	6.43	1.859	-18.7

From the Van't Hoff plot [ln(Kd) Vs 1/ T], the thermodynamic parameters were determined:

- $\Delta H°$ = +12.4 kJ/mol
- $\Delta S°$ = +95.2 J/mol·K
- R^2 = 0.997

4.2.4. Adsorption Kinetics : The kinetic data were fitted to pseudo-first-order and pseudo-second-order models. The pseudo-second-order model showed better correlation ($R^2 > 0.99$) for all temperatures studied, indicating that chemisorption is the rate-limiting step.

4.2.5. Equilibrium Isotherms : Langmuir and Freundlich isotherm models were applied to the equilibrium data. The Langmuir model provided better fit with maximum adsorption capacity (qmax) of 85.2 mg/g at 298 K and Langmuir constant (KL) of 0.089 L/mg.

V. DISCUSSION

The negative ΔG° values at all temperatures confirm the spontaneous nature of Fluorescein Sodium adsorption on bentonite. The increasingly negative ΔG° values with rising temperature indicate enhanced spontaneity at higher temperatures, which is consistent with the observed increase in removal efficiency.

The positive ΔH° value (+12.4 kJ/mol) indicates that the adsorption process is endothermic, explaining the improved adsorption capacity at higher temperatures. This suggests that the adsorption mechanism involves bond formation between dye molecules and bentonite surface, requiring energy input to overcome activation barriers.

The positive ΔS° value (+95.2 J/mol·K) indicates increased randomness at the solid-liquid interface during adsorption. This entropy increase can be attributed to the displacement of water molecules from the bentonite surface upon dye adsorption and structural changes in the clay layers. The magnitude of ΔH° suggests that the adsorption involves physical interactions rather than strong chemical bonding, as chemical adsorption typically exhibits ΔH° values greater than 20 kJ/mol. The combination of van der Waals forces, electrostatic interactions, and hydrogen bonding likely governs the adsorption mechanism.

VI. CONCLUSION

This comprehensive study successfully demonstrated the effectiveness of bentonite clay as an adsorbent for Fluorescein Sodium dye removal from aqueous solutions. The key findings include:

1. High Removal Efficiency: Bentonite achieved maximum removal efficiency of 90.6% at 328 K with an adsorption capacity of 85.2 mg/g, demonstrating its potential as an effective adsorbent.
2. Spontaneous Adsorption: The negative Gibbs free energy values (-15.8 to -18.7 kJ/mol) confirmed the thermodynamically favorable and spontaneous nature of the adsorption process across all temperatures studied.
3. Endothermic Nature: The positive enthalpy change (ΔH° = +12.4 kJ/mol) indicated that the adsorption process is endothermic, with higher temperatures favoring increased dye removal.
4. Entropy-Driven Process: The positive entropy change (ΔS° = +95.2 J/mol·K) suggested increased randomness at the solid-liquid interface, contributing to the spontaneity of adsorption.
5. Physical Adsorption Mechanism: The relatively low enthalpy value indicates that physical interactions, including van der Waals forces and electrostatic attractions, primarily govern the adsorption process.
6. Optimal Conditions: The study identified optimal operating conditions as 328 K temperature, pH 6, 180 minutes contact time, and 1.5 g/L adsorbent dosage for maximum removal efficiency.
7. Kinetic and Equilibrium Behavior: The adsorption followed pseudo-second-order kinetics and fitted well with the Langmuir isotherm model, indicating monolayer adsorption on homogeneous surfaces.

The results provide valuable insights into the thermodynamic feasibility of using bentonite for dye removal applications and contribute to the development of sustainable wastewater treatment technologies. The positive thermodynamic parameters and high removal efficiency make bentonite a promising candidate for industrial-scale dye removal processes. Future research should focus on investigating the regeneration potential of bentonite, optimization process parameters for continuous flow systems, and evaluation of treatment effectiveness for real industrial wastewater containing multiple dyes and contaminants.

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